I CLAIM:

1. A method of speciated isotope dilution measurement of a sample comprising

providing at least one predetermined stable isotope,

preparing an isotopic spike by converting said stable isotope to a speciated enriched isotope corresponding to the species to be measured in said sample,

spiking the sample containing said specie to be measured,

equilibriating said isotopic spiked species with said species to be measured,

separating at least a portion of said species from said sample,

making isotope ratio determinations for each said specie to be measured and mathematically deconvoluting said species concentration while correcting for species conversion, and

effecting said mathematical deconvolution while correcting for incomplete separation of said species from said sample.

2. The method of speciated isotope dilution measurement of claim 1 including

employing said method on more than one said species to be measured simultaneously.

3. The method of speciated isotope dilution measurement of claim 2 including

employing said method in quantifying Cr(III) and Cr(VI).

4. The method of speciated isotope dilution measurement of claim 2 including

effecting said mathematical deconvolution employing the following formulas:

$$R^{III}_{50/52} = \frac{({}^{50}A_x C_x^{III} W_x + {}^{50}A_s^{III} C_s^{III} W_s^{III})(1-\alpha) + ({}^{50}A_x C_x^{VI} W_x + {}^{50}A_s^{VI} C_s^{VI} W_s^{VI})\beta}{({}^{52}A_x C_x^{III} W_x + {}^{52}A_s^{III} C_s^{III} W_s^{III})(1-\alpha) + ({}^{52}A_x C_x^{VI} W_x + {}^{52}A_s^{VI} C_s^{VI} W_s^{VI})\beta}$$

$$R^{III}_{53/52} = \frac{({}^{53}A_x C_x^{III} W_x + {}^{53}A_s^{III} C_s^{III} W_s^{III})(1-\alpha) + ({}^{53}A_x C_x^{VI} W_x + {}^{53}A_s^{VI} C_s^{VI} W_s^{VI})\beta}{({}^{52}A_x C_x^{III} W_x + {}^{52}A_s^{III} C_s^{III} W_s^{III})(1-\alpha) + ({}^{52}A_x C_x^{VI} W_x + {}^{52}A_s^{VI} C_s^{VI} W_s^{VI})\beta}$$

$$R^{VI}_{50/52} = \frac{({}^{50}A_x C_x^{III} W_x + {}^{50}A_s^{III} C_s^{III} W_s^{III})\alpha + ({}^{50}A_x C_x^{VI} W_x + {}^{50}A_s^{VI} C_s^{VI} W_s^{III})(1-\beta)}{({}^{52}A_x C_x^{III} W_x + {}^{52}A_s^{III} C_s^{III} W_s^{III})\alpha + ({}^{52}A_x C_x^{VI} W_x + {}^{52}A_s^{VI} C_s^{VI} W_s^{VI})(1-\beta)}$$

$$R^{VI}_{53/52} = \frac{({}^{53}A_x C_x^{III} W_x + {}^{53}A_s^{III} C_s^{III} W_s^{III})\alpha + ({}^{53}A_x C_x^{VI} W_x + {}^{53}A_s^{VI} C_s^{VI} W_s^{III})(1-\beta)}{({}^{52}A_x C_x^{III} W_x + {}^{52}A_s^{III} C_s^{III} W_s^{III})\alpha + ({}^{52}A_x C_x^{VI} W_x + {}^{52}A_s^{VI} C_s^{VI} W_s^{VI})(1-\beta)}$$

where,

$R^{III}_{50/52}$ is the measured isotope ratio of ^{50}Cr to ^{52}Cr of Cr(III) in the spiked sample

$^{50}A_x$ is the natural atomic fraction of ^{50}Cr in the sample

C_x^{III} is the concentration of Cr(III) in the sample (μmole/g, unknown)

W_x is the weight of the sample in grams

$^{50}A_s^{III}$ is the atomic fraction of ^{50}Cr in the isotopic spike: ^{50}Cr(III)

C_s^{III} is the concentration of Cr(III) in the ^{50}Cr(III) spike (μmole/g)

W_s^{III} is the weight of the ^{50}Cr(III) spike in grams

C_x^{VI} is the concentration of Cr(VI) in the sample (μmole/g, unknown)

α is the percentage of Cr(III) oxidized to Cr(VI) after spiking (unknown)

ß is the percentage of Cr(VI) reduced to Cr(III) after spiking (unknown)

5. The method of speciated isotope dilution measurement of claim 1 including

employing a mass spectrometer to determine said isotopic element ratios.

6. The method of speciated isotope dilution measurement of claim 1 including

tagging said enriched isotope with an isotopic tag in the same speciated form as the species to be measured.

7. The method of speciated isotope dilution measurement of claim 1 including

employing time resolution chromatography to effect said separation.

8. The method of speciated isotope dilution measurement of claim 1 including

employing said process on a sample which has experienced specie conversion prior to separation.

9. The method of speciated isotope dilution measurement of claim 1 including

effecting said equilibrium in an aqueous solution.

10. The method of speciated isotope dilution measurement of claim 1 including

employing said process on an incompletely separated specie.

11. The method of speciated isotope dilution measurement of claim 3 including

effecting said separation after reduction of a substantial portion of Cr(VI) to Cr(III).

12. The method of speciated isotope dilution measurement of claim 1 including

employing said process on a soil sample.

13. The method of speciated isotope dilution measurement of claim 1 including

employing said process on an aqueous sample.

14. The method of speciated isotope dilution measurement of claim 1 including

employing said process on solid waste from a chromite ore processing system.

15. The method of speciated isotope dilution measurement of claim 1 including

storing said sample after said equilibrating step and prior to said separating step.

16. The method of speciated isotope dilution measurement of claim 1 including

effecting said mathematical deconvoluting simultaneously with respect to more than one species to be measured.

17. The method of speciated isotope dilution measurement of claim 16 including

effecting said mathematical deconvoluting for each species independently of other species.

18. The method of speciated isotope dilution measurement of claim 1 including

effecting said mathematical ~~deconvoulting~~ deconvoluting with respect solely to one species to be measured.

19. The method of speciated isotope dilution measurement of claim 16 including

effecting said separation of at least about 5 to 10 percent, but less than 100 percent of each said species from said sample and from said other species before effecting said deconvolution.

20. The method of speciated isotope dilution measurement of claim 1 including

effecting said separation by at least one method selected from the group consisting of chromatography, microwave assisted extraction, soxhilate extraction, solvent dissolution, acid dissolution, acid or base hydrolysis distillation, centrifugation, and solvent extraction.

21. The method of speciated isotope dilution measurement of claim 1 including

effecting said separating by microwave assisted extraction for a period of about 5 to 15 minutes.

22. The method of speciated isotope dilution measurement of claim 21 including

effecting said separating at about 90°C to 150°C.

23. The method of speciated isotope dilution measurement of claim 1 including

after effecting said speciated isotope dilution measurement of a sample comparing the results of said measurement with measurements made by another type of test to evaluate the validity of said another type of test.

24. The method of speciated isotope dilution measurement of claim 23 including

effecting a plurality of measurements by said another test and effecting said comparison in evaluating the validity of said another type of test.

25. The method of speciated isotope dilution measurement of claim 1 including

employing said method to prepare speciated spiked standard materials.

26. The method of speciated isotope dilution measurement of claim 1 including

employing said method to prepare standard materials.

27. The method of speciated isotope dilution measurement of claim 25 including

creating said speciated spiked standard by spiking separated stable isotopes in speciated form.

28. The method of speciated isotope dilution measurement of claim 26 including

employing said standard materials after storage.

29. The method of speciated isotope dilution measurement of claim 26 including

employing said method to correct species shifts in said standard materials after degradation.

30. The method of speciated isotope dilution measurement of claim 21 including

employing in said microwave extraction closed vessel microwave extraction.

31. The method of speciated isotope dilution measurement of claim 23 including

employing said method to validate said tests which are not independently capable of compensating for incomplete species extraction or species conversion.

32. The method of speciated isotope dilution measurement of claim 1 including

performing said method on a species which due to incomplete separation, loss, conversion or degradation has less than 100 percent of said species separated.

33. The method of speciated isotope dilution measurement of claim 32 including

effecting said separation of at least about 5 to 10 percent of said species.